Metallurgy Department Annual Progress Report for the Period Ending March 31st, 1966

Forskningscenter Risø, Roskilde

Publication date:
1966

Document Version
Publisher's PDF, also known as Version of record

Risø Report No. 139

Danish Atomic Energy Commission

Research Establishment Risø

Metallurgy Department
Annual Progress Report

for the Period Ending March 31st, 1966

September, 1966

Sales distributors: Jul. Gjellerup, 87, Sölvgade, Copenhagen K, Denmark
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Danish Atomic Energy Commission
Research Establishment Risö

METALLURGY DEPARTMENT

ANNUAL PROGRESS REPORT

for the Period Ending
March 31st, 1966

ABSTRACT

The Department's research work in reactor metallurgy was centred around problems connected with fuel elements for power and research reactors and steels for reactor pressure vessels.

The fuel-element work was concerned with SAP/uranium-dioxide and zircaloy/uranium-dioxide elements for power reactors, cooled with organic liquid and water respectively, as well as plate fuel elements for research reactors.

The experiments with the SAP/UO_2 prototype element for the Risø study project DOR are completed in part. The experimental work is continuing with investigations of the irradiation properties of SAP/UO_2 fuel pins; twenty pins of lengths from 10 to 15 cm have been irradiated in DR3 and are being examined in the hot-cell facility. With the irradiation of a further ten pins this programme will be completed.

The work with SAP material has been of interest in connection with the revival in the U.S.A. of work on the organic-cooled reactor, and a continuous exchange of results with American research laboratories has taken place.

The experiments with zircaloy/UO_2 fuel elements are carried out in co-operation with the Elsinore Shipbuilding and Engineering Co., Ltd., with a view to industrial production of fuel elements in Denmark. As the first phase, a series of elements will be manufactured for irradiation in the Halden reactor in Norway. A specification for zircaloy materials has been prepared, and initial experiments on welding, non-destructive quality control and corrosion testing have been carried out.

Within the fields of welding and non-destructive testing, the Department co-operates with the Danish Central Welding Institution, which, especially in the latter field, has been able to utilise its experience from corresponding work with SAP.

The research work with UO_2 included experiments on precipitation, pressing and sintering of pellets in order to improve their quality and reproducibility. Pellets irradiated in DR3 are being examined in the hot cells with particular regard to fission-gas release, volume increase and structural changes.

The experimental manufacture of plate-shaped fuel elements was continued in co-operation with the Elsinore Shipbuilding and Engineering Co., Ltd. The first fuel element was loaded into DR3 in December 1965 and has

since been followed by two more. The elements have performed satisfactorily during irradiation, and the fabrication process will now be tested further by the manufacture of a large number of elements.

In connection with the Dano-Swedish co-operation in the reactor field a joint research programme has been started, involving the investigation, before and after neutron irradiation, of high-strength reactor pressure-vessel steels. The irradiation experiments will be carried out in DR3 at Risö and in R3 at Studsvik, Sweden. In connection with this work, a group with representatives of Burmeister and Wain, Engineers and Shipbuilders Limited, the Central Welding Institution and the Metallurgy Department has begun an evaluation of what research will be necessary, especially within the fields of welding and inspection, as a basis for reactor pressure-vessel manufacture in Denmark.

The experimental work under the agreement with the U. K. A. E. A. concerning investigations of uranium and uranium alloys before and after neutron irradiation was concluded in March 1966. The experimental results will be published together with supplementary results obtained in England.

In connection with the co-operation agreement with the U. K. A. E. A. concerning fundamental material research, especially on the reactor irradiation behaviour of materials and electron microscopy, a scientist from the British research establishment at Harwell was exchanged for one from the Department. The research comprised, among other subjects, irradiation damage in aluminium - magnesium alloys.

The co-operation with the U. K. A. E. A. has been extended so as to include problems related to the use of zirconium alloys, and a scientist from the U. K. A. E. A. has been seconded to the Department for a period of about eighteen months to work on various subjects, including joint programmes involving dispersion-strengthened zirconium materials with good high-temperature strength. This research programme is based upon the U. K. A. E. A.'s experience of zirconium materials and the Department's experience of dispersion strengthening obtained in connection with the SAP work.

The experimental work of a general metallurgical character included the development of dispersion-hardened, high-temperature-resistant aluminium materials. A series of alloys was manufactured, and the influence of variables such as grain size, particle distribution and structure, and deformation on the mechanical properties of the materials was studied. An investigation into the ductility of these alloys was carried out by a post-graduate student in preparation for his acquisition of the lic. techn. degree.

Other experiments were concerned with texture studies of aluminium/aluminium-oxide materials, fractionating of aluminium powders (carried out in co-operation with the Metallurgy Laboratory at the Technical University of Copenhagen), sintering of aluminium powder, and preparation of aluminium/aluminium-oxide single crystals. As a lead in this work, a continuous literature survey, released in the form of Risö Reports, is carried out in co-operation with the Risö Library.

Among general metallurgical experiments may also be mentioned the following: continued development of methods for welding steel-encapsulated thermocouples, about 1800 of which were manufactured for the departments of Risö and for various industrial concerns; texture studies of face-centred cubic metals in order to establish the relationship between degree of deformation, texture and mechanical properties; investigation of the influence of cold pressing on the density of sintered powder specimens; electrochemical experiments concerning the effectiveness of corrosion inhibitors; dislocation formation during quenching, and fission fragment damage, in aluminium alloys.

For Danish industrial concerns work was performed on a number of problems in the fields of rolling, casting, gas analysis, X-ray diffraction, sintering, welding, soldering, corrosion, and material testing. With a view to making its results available to Danish industry, the Department participates in a working party set up by the Research Committee of the Federation of Danish Mechanical Engineering and Metal-Working Industries. In connection with this work, monthly articles about metallurgical techniques and apparatus are published in the Danish engineers' weekly news journal. Furthermore, a member of the Department has been given a seat on the Corrosion Board, which acts as an advisory organ for the Corrosion Centre of the Danish Academy of Technical Sciences.

Detailed information about the activities of the Metallurgy Department is set out in its annual progress report; information is also given in the form of quarterly progress reports.

CONTENTS

MATERIAL PROPERTIES

Uranium-Dioxide/Zircaloy Fuel Elements for Water Reactors

In July 1965, water-reactor fuel technology was taken up, the start being made with a project aimed at developing a fabrication route for UO_2/zircaloy fuel elements. The results of the work are intended, as far as possible, to be applicable to light-water (PWR and BWR) as well as heavy-water (BHWR) reactors. A route for the manufacture of UO_2 pellets had already been established in connection with the DOR[1] project, and some of the SAP technology should also be applicable, notably that of non-destructive testing.

An assessment of the possible irradiation facilities pointed to the Halden BHWR as the most suitable, and a development programme was drawn up based upon the mounting of irradiation experiments in the 3rd core of that reactor, commencing in July 1967. The specifications for the first experiment correspond to the Halden 3rd core design, with peak heat ratings equal to those of the current DK 400 design[2] (610 W/cm). The experiment will essentially be a test of fuel pins manufactured at Risø, but one assembly will incorporate some variations in pellet geometry and pellet can clearance aimed at elucidating their effects on pellet and can behaviour. In later experiments it is intended to incorporate more advanced concepts and designs from the DK 400 project.

The work being carried out and envisaged under the above programme comprises:

1. Inspection, including non-destructive testing.
2. Fabrication of UO_2 fuel.
3. Fabrication of fuel pins, in particular welding and autoclaving.
4. Evaluation of cladding by chemical analysis, metallography, mechanical testing, and corrosion testing.
5. Supporting irradiation experiments in DR3.
6. Post-irradiation examination.

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1) DOR was a Risø assessment study of an organic-cooled, heavy-water-moderated reactor.

2) DK 400 is a Risø assessment study of a boiling-heavy-water reactor (BHWR).

The necessary plant modifications and arrangements for the provision of new plant are in hand, and an initial supply of zircaloy-2 cladding tubing and end plug bars was received in December. After inspection and non-destructive testing this material is being used for preliminary fabrication and testing development. In the meantime detailed specifications for zircaloy tubes and bars have been drawn up, and enquiries preparatory to purchasing material for the main programme have been issued.

In order to keep abreast of water-reactor fuel developments a development programme has been drawn up covering the period from 1966 to 1969. The main subjects of development proposed are

1. UO_2 fuel fabrication processes, including vibratory compaction.
2. Continued evaluation of zircaloy cladding and evaluation of alternative materials.
3. Improved non-destructive testing techniques for cladding tubes and fuel pins.
4. Improved fuel-pin fabrication techniques, particularly welding.
5. Post-irradiation examination techniques.

Where appropriate, the investigations will include irradiation testing in DR3, Halden or another facility.

Material Testing and Metallography

Metallography of Zircaloy-2

The metallographic method of preparing specimens of zircaloy-2[3] comprises a normal wet pregrinding on silicon-carbide papers, followed by a prepolish with 7-micron diamond paste. The final polishing is carried out as an attack polish with CrO_3 and distilled water. This treatment gives a scratch-free surface and a good contrast for examining general structure and grain sizes in polarized light (fig. 1 left).

For examination of hydrides the specimens are swab etched in a solution of 25 ml C_2H_5OH, 10 ml conc. HNO_3, 50 ml conc. H_2O_2, and 4 ml conc. HF, which reveals the hydrides clearly (fig. 1 right) and at the same time allows structural examinations in polarized light.

Flare Testing of Zircaloy Tubes

In most specifications for zirconium tubes the flare test[4] is prescribed as a rapid and simple multiaxial ductility test. The test uses a hardened steel core of 60° included angle which is pushed into the end of

3) Journ. Inst. Metals 94 (7), 245 (1966).

4) A.S.T.M. Designation B. 353-62 T (1962).

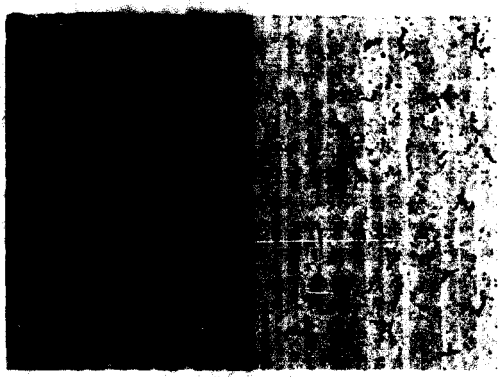

Fig. 1. Zircaloy-2 etch polished. The left picture shows the grain structure in polarized light, the right one shows hydride precipitation after etching. 300 x.

the tube until the diametral expansion exceeds a specific value, which must be achieved without any cracking or splitting of the tube. At the laboratory the flare test is used for zircaloy-2 tubes which - both in the as-received condition and after autoclaving for three days at 400°C - should be able to withstand a diametral expansion of $33\frac{1}{3}$% without failure. The test, results of which are shown in fig. 2, gives a certain guarantee against accepting tubing with radial hydride orientation tendencies, which may reduce the ductility of the tube.

Fig. 2. Zircaloy-2 tubes after a "flare test". The left tube has had a diametral expansion of 33 1/3% without failure, while the right tube has been tested until cracking started at 45% diametral expansion.

Hydrogen Determination in Zircaloy-2

The apparatus for gas analysis, built at the laboratory, was used for the determination of hydrogen in zircaloy-2 by the hot-extraction method. The specimens of zircaloy-2 were heated in a graphite crucible at 1250°C in vacuum. The hydrogen, which is the only gas evolved at that temperature[5], is transferred from the heating zone with a mercury diffusion pump to a calibrated volume, where the amount of hydrogen is determined by measurement of the pressure with a McLeod gauge. The results obtained show good agreement with results from the "Sentral Institutt for Industriel Forskning" in Norway, as appears from table I

Table I

Hydrogen Determination in a Zircaloy-2 Rod

Sample no.	Hydrogen content in ppm				
	1	2	3	4	Average
Sentralinstitutt for Industriel Forskning, Norway	3.6	3.5	3.4	3.5	3.5
Metallurgy Department, Risø, Denmark	3.4	3.3	3.0	3.3	3.3

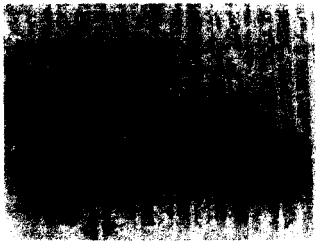

Fig. 3. Test section after burst of tube.

5) R. K. McGeary, "Determination of Hydrogen in Zirconium by the Hot Extraction Method" (ASM, Cleveland, 1953) 168-175.

Examination of Failed SAP Tube

For examination of the joints of SAP and steel tubes a small rig has been set up allowing these joints to be tested under static conditions at 400°C and with an internal pressure of max. 15 kg/cm^2. After being tested for 2200 hours at 400°C, a SAP tube burst. The tube (fig. 3) was delivered early in 1960 and had an oxide content of 10%, an inside diameter of 96 mm and a wall thickness of 5 mm. The microstructure of the tube was heavily damaged by a great number of large cracks and holes (fig. 4). In order to investigate the structural stability of the tube material at higher temperatures, some as-delivered tube material was heat treated and analysed for gas content. The treatment produced a great number of cracks and holes in the microstructure, and a large hydrogen content (55 ppm) was found in the as-delivered material.

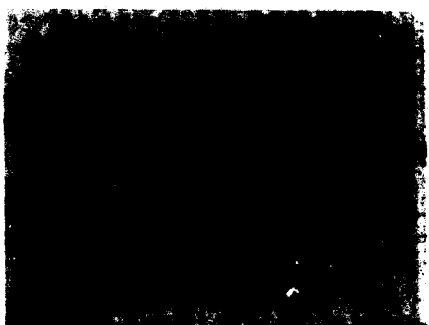

Fig. 4. Longitudinal section of the failed tube outside the crack, unetched. 500 x.

As the pressure applied to a tube during its lifetime is smaller than that required to burst the tube in identical circumstances, the cause of the failure must be the structural instability of the material at high temperatures. The combination of stress, temperature and high gas content leads to the formation of numerous cracks and holes in the material, which reduce its strength and finally make it burst.

High-Temperature Materials

Inconel 600 and incoloy 800 alloys, which have been used in production facilities, were examined with respect to the strength of the materials at different temperatures and possible changes in their microstructure.

Fig. 5. Springfields adjusted uranium after 700 MWD/te, attack polished, showing a mottled structure. 90 x.

Remote Metallography

The remote metallography consisted of investigations of irradiated uranium alloys and irradiated uranium dioxide and SAP. The irradiated uranium and uranium-alloy specimens were prepared by wet pregrinding with alcohol as liquid, followed by diamond polishing and attack polishing in alumina and conc. hydrogen peroxide. The method gave a good contrast in polarized light and a scratchfree surface. All the specimens examined had a very mottled structure as illustrated in fig. 5, which shows adjusted Springfields uranium in polarized light.

Fig. 6. UO_2 pellet irradiated to 1000 MWD/te with a centre temperature of approximately 1100°C, etched. 800 x.

The uranium-dioxide specimens were prepared by the same method as the uranium alloys, including the attack polish. After the polishing operations the specimens were etched in a mixture of H_2O_2 and H_2SO_4; the results are shown in fig. 6.

The irradiated SAP material was prepared for microscopic investigation by wet pregrinding followed by prepolishing with diamonds (5 and 7 microns) and vibratory polishing with alumina for three hours. The resulting structure shows a scratchfree surface with no changes due to the irradiation (fig. 7).

Fig. 7. SAP-986 after irradiation at 400°C with a dose of approximately 5×10^{20} n/cm^2 (E > 1 MeV). 80 x.

Corrosion

Electrochemical Corrosion Studies

The electrochemical equipment was used for potentiostatic and potentiodynamic experiments, especially on the effect of corrosion inhibitors in industrial cooling water. Most experiments were concerned with mixtures of chromate and polyphosphate, but also a few other inhibitors were involved. The concentrations of inhibitors, pH and oxygen in the water were used as parameters. It was attempted to calculate the corrosion rates on the basis of the polarization curves, and the absolute as well as the relative corrosion rates were in reasonably good agreement with long-term weight-change measurements.

A logarithmic converter has been developed by the Electronics Department to be put in between the potentiostat and the X-Y recorder. This equipment has been run in, and it is now possible to get a direct record of the logarithm of the current versus the potential.

Corrosion of Zircaloy-2

In connection with the UO_2/zircaloy fuel-element programme the problems of oxidation and corrosion testing of zircaloy-2 have been taken up. The corrosion testing and the oxidation are carried out in superheated steam or high-temperature water, high- or low-pressure autoclaves being used. Equipment for the testing has been procured, and literature studies and planning have been carried out.

The main points are:

1) Testing of incoming material.

The corrosion resistance of the incoming material is checked by a short-term test (superheated stem, 400°C, 100 psi, 3 days) and a long-term test (superheated steam, 400°C, 1500 psi, several months). Equipment has been procured for short-term tests, and the testing has been started. For the long-term, high-pressure tests a high-pressure autoclave has been borrowed from the Technical University of Denmark and is under commissioning. In connection with the autoclaving, studies on the pretreatment (pickling etc.) of the zircaloy have been carried out.

2) Testing of welds.

As the welding zones are very susceptible to contamination for instance from an unclean welding atmosphere, resulting in a reduced corrosion resistance, corrosion testing is carried out in close connection with the welding programme. The testing is performed in the same way as the above-mentioned testing of incoming material; the short-term testing has been started.

3) Conditioning of tubes for fuel elements

To give the tubes for fuel-element fabrication the most corrosion-resistant surface and to condition them for further hardening they are usually autoclaved in low-pressure superheated steam at 400°C for three days. A project for construction of an autoclave (length 4 m) for this purpose has been started in co-operation with the Engineering Department at Risø.

Uptake by SAP of Hydrogen from Terphenyls

The experiments on the uptake by SAP of hydrogen from terphenyls (Progil OM2) in autoclaves have been finished. The experiments were carried out at 400 and 450°C, and the principal parameter was the water content in the terphenyl. Water (up to 2000 ppm) was added in small glass ampoules

which broke during the heating of the autoclaves. The hydrogen content in the specimens was determined by gas analysis and compared with the hydrogen content in the material as delivered. For the analysis, the specimens were melted in a molten-iron bath in an induction-heated graphite crucible, the released gas was pumped off in a vacuum system, and the amount of gas was determined.

Experimental results are given in table II. They have to be compared with the hydrogen content in the original material, 10-12 ppm, determined by the same method. In spite of the considerable spread of the results it is obvious that the increase in hydrogen content, if any, is very slight, and that the water content in the terphenyl is of no importance.

Table II

Uptake by SAP of Hydrogen from Terphenyls

Temp.	Added H_2O ppm	Duration hours	ppm H_2 in SAP		
400°C	0	336	13.7		13.9
		1440	12.4		16.1
	200	336	8.4		16.6
		1440	27.0		27.1
	500	336	11.0		11.1
		1440	15.4		25.0
	2000	336	11.0		14.0
		1440	13.3		14.0
450°C	0	336	7.2		9.0
			11.0		13.7
			15.5		
		1440	13.4		13.9
	200	336	12.6		17.2
	500	336	18.0	18.3	17.3
			18.0	9.5	8.6
			8.2	7.2	
		1440	9.8	17.8	12.6
			13.6		
	2000	336	10.5	11.3	8.5
		1296	16.2		18.9
		1440	13.9		14.3

Corrosion of DR2 Fuel Element

A standard fuel element (MTR-type) from DR2 had been stored in demineralized water for more than five years and looked badly corroded when it was inspected in the hot cells. The type of corrosion and the corrosion products were investigated in detail, and the total amount of corrosion was estimated. The bad appearance of the fuel element was found to be due

to the scaling off of a very thin layer of corrosion products when the element dried up. The amount of corrosion products corresponded to a weight gain of approximately 100 mg/dm^2, and the distribution of the attack was uniform. The weight gain corresponds to a total corrosion of about 2 microns, which is quite normal for aluminium after prolonged storage in pure water.

Corrosion of Stainless Steel in Boiling Concentrated Sea Water

The long-term experiments on the development of pitting in austenitic stainless steel (AISI 316) in boiling, concentrated sea water with and without air continued during the whole period.

Boiling Heavy-Water Reactor Project (DK 400)

The Metallurgy Department co-operated with the Chemistry Department in studies on water treatment and the use of boric acid as a burnable poison in the DK 400 project.

Corrosion of Steel-Aluminium Cable

An investigation of the causes of severe corrosion damage to a steel-aluminium cable for electric power transmission was carried out.

Physical Metallurgy

Texture of Extruded Aluminium/Aluminium-Oxide Alloys

The texture of different SAP-type and powder-blended Al/Al_2O_3 alloys was measured quantitatively with a Schulz texture goniometer. For all the alloys investigated, except the higher-oxide SAP types, a dual ⟨100⟩ - ⟨111⟩ fibre texture was found. The higher-oxide SAP alloys showed only ⟨111⟩ texture.

For the powder-blended alloys made from coarse aluminium powder by extrusion at 500°C, the two texture components are of about equal strength, whilst the material made from powder with finer grains has about 80% ⟨111⟩ and 20% ⟨100⟩. In the powder-blended alloys the oxide content and the grain size can be varied independently, and it has been found that the texture, as described by the amounts of material having the ⟨111⟩ and the ⟨100⟩ direction respectively within 15° from the extrusion direction, is independent of the oxide content and only determined by the grain size, whereas the sharpness of the texture is governed by the oxide content.

In the powder-blended alloys, practically all grains conform to one of the two textures. In the SAP alloys of low oxide content a small part, in the higher-oxide SAP alloys a larger part, of the grains are randomly oriented.

The microtexture was studied by electron microscopy with special attention to a possible morphological difference between the ⟨100⟩ grains and the ⟨111⟩ grains, but it was not possible to detect such a difference. This indicates that, for these alloys, the ⟨100⟩ texture component is not a recrystallization texture as normally stated, but a deformation texture. This is further supported by the fact that annealing of the extruded alloys at temperatures where grain growth occurs, makes no change in the ⟨100⟩ / ⟨111⟩ ratio. If the ⟨100⟩ texture were a recrystallization texture, one would expect to find preferential growth of the ⟨100⟩ grains.

Rolling Textures in Face-Centred Cubic Metals and Alloys

By deformation of polycrystalline materials with random crystal orientation a preferred orientation (a texture) will develop, caused by overall grain rotations the nature of which depends on the deformation modes of the material.

On rolling of face-centred cubic metals and alloys, the resulting texture is one of the two types classified for convenience as copper type and brass type.

These different preferred orientations, developed under similar conditions by copper and brass of the same crystal structure, must be attributed to differences in the deformation characteristics.

Various theories have been put forward to enable these two types of rolling texture to be predicted from a knowledge of the deformation modes of the metals. The three most reasonable theories seem to be the cross-slip theory, the mechanical twinning theory and the theory of slip in other planes than {111}.

This project is an attempt to clarify the problems of the two texture types.

The materials used for the experiments were copper and zinc of a purity of 99.99%. Melting and casting of copper and brass with 15% zinc were carried out in an induction furnace employing an atmosphere of argon. The material was initially deformed 50% and homogenized at 550°C, which resulted in a material as "texture-free" as possible with a grain size of about 0.03 mm. The materials were rolled at room temperature to 98% re-

duction in thickness. In order to investigate the texture development during the rolling, samples were taken of different reductions and prepared for X-ray examination by removing half the thickness of the rolled sheets by machining. After electropolishing, pole figures were determined with a Schulz texture goniometer.

From the pole figures for copper and brass it appears that the texture characteristic of the two types is already developed at 65-80% rolling reduction in thickness, whilst this texture is normally stated to be developed only at about 95% reduction.

Hydrogen in Aluminium/Aluminium-Oxide (SAP)

Determination of the hydrogen development in sintered Al/Al_2O_3 during heating was attempted. Although the experiments yielded reasonably reproducible values for the total amount of hydrogen extracted, the accuracy of the extraction was somewhat unsatisfactory. This was attributed to inadequacy of the vacuum equipment. Consequently, high-vacuum apparatus with a negligible leak rate has been procured, which opens up prospects of extending the present experiment into a more general gas-diffusion analysis.

Fission-Fragment Damage in Dilute Aluminium-Magnesium Alloys

A staff member visiting the Metallurgy Division, A. E. R. E., Harwell, England, made a study of the effect of magnesium concentration (up to 0.5 a/o) on the formation of dislocation loops in aluminium during fission-fragment bombardment at 60°C. Increasing magnesium content reduced the initial rate of increase of the total loop area with dose, but the loop area was found to be saturated at a higher level in more concentrated alloys. A magnesium concentration as low as 0.02% reduced the initial rate of loop growth by a factor of 4 to 5. These results were explained in terms of trapping effects on the mobility of interstitial atoms and vacancies. The aluminium foils were irradiated in the pre-thinned state and the measurements carried out by means of electron microscopy. Considerable attention was given to the problem of obtaining accurate quantitative results, and several corrections, e.g. for foil tilt, surface denudation and contrast conditions, were applied.

Preliminary attempts to examine the mechanisms of cluster formation in more detail by varying the irradiation temperatures were made at A. E. R. E., Harwell; however, the analyses mainly took place at Risö. The results have not been conclusive so far, and the experiments will be continued at Risö.

Single Crystals of Aluminium/Aluminium-Oxide Alloys*

Experiments on the production of single crystals of Al/Al_2O_3 were moderately successful. Flat, rolled specimens were unable to give single crystals by the strain - annealing method. A 1 mm wire of MD 13 (about 0.05 vol. % Al_2O_3) was drawn 10% and passed through a vertical furnace. By the X-ray Laue method the wire was verified to consist of two crystals in cross section, each of them about 60 mm long. The two crystals were probably caused by the grips used when drawing the wire.

Mechanical Properties in Relation to Structure of Aluminium/Aluminium-Oxide Alloys

The investigations of the effect of the grain size of the aluminium matrix and of the oxide distribution (oxide particle size, oxide particle spacing and vol. % of oxide) were continued, but no definite conclusions regarding the strengthening mechanism were obtained. The difficulties are mainly caused by the difference between the available theories, which are concerned with single crystals with uniform oxide distribution, and the experimental polycrystalline alloys with a rather non-uniform oxide distribution. Furthermore the strengthening parameter in the theories is mainly the dislocation density, whilst the experimentally measurable parameters are grain size and oxide content, and possibly oxide particle spacings. A correlation between these seems necessary.

Thickness Measurement of Specimens for the Electron Microscope*

Specimens of Al/Al_2O_3 alloys for the electron microscope are prepared by combined electropolishing and chemical polishing. It is important that the specimens are 60-80 microns before the chemical polishing. An apparatus has been constructed for measurement of the thickness of the electropolished, disc-shaped specimens. The specimen in the holder (PTFE-holder) is placed near to a focussed beam of β-rays (Tl 204 isotope), and the transmitted beam is caught by a Geiger detector and fed to a count-rate meter. A calibration curve relates the reading to the thickness (the half-life of the Tl 204 is 4.1 years so that adjustment of the curve is only rarely needed). The measurement is accurate to within $\pm$ 10 microns.

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* This work was carried out in preparation for a thesis for the Technical University of Denmark.

Creep Properties of Polygonized Alloys, Especially Aluminium/Aluminium-Oxide Products

Alloys of MD 13 (0.06 wt. % oxide) and of MD 105 powder (0.6 wt. % oxide) were made for creep experiments. The microstructure of these materials consists of a finely distributed oxide phase and a matrix structure divided into grains. The experiments were performed at 400°C with loads to give creep rupture times from 1 to 1000 hours. The grain size of these products is varied by deforming the materials to different degrees and recrystallizing, while the oxide distribution is varied by re-extruding an extruded rod in a direction perpendicular to the original one.

Dispersion-Strengthened Zirconium Products

A programme has been started to investigate the possibilities of applying dispersion strengthening to zirconium.

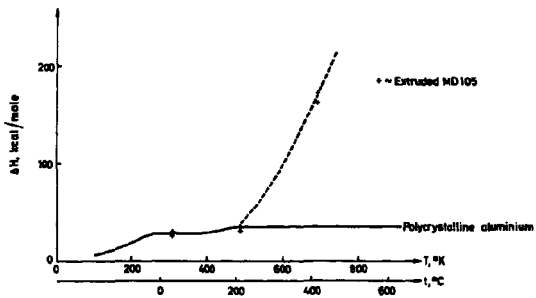

Fig. 8. Activation energies in creep of aluminium alloys.

Activation-Energy Determination by Deformation of an Aluminium/Aluminium-Oxide Alloy[K]

Activation energies were determined between 21 and 400°C by creep testing of an Al/0.6 wt. % Al_2O_3 alloy, called MD 105, Dorn's method[6] being used. Fig. 8 shows the activation energies in the extruded alloy at around

[K] This work was carried out in preparation for a thesis for the Technical University of Denmark.

[6] J. E. Dorn, Creep and Recovery. ASM symposium (ed. by Maddin), Cleveland, 1957, p. 255.

21, 200 and 400°C compared with creep activation energies in pure Al[7]. At 21 and 200°C the values are the same, whilst the activation energy in the Al/Al_2O_3 alloy at high temperature is greater than the constant value of 36 kcal/mole in pure Al. Activation energies were also determined by tensile testing of both extruded fine-grained and recrystallized coarse-grained MD 105 alloy at 300, 400 and 500°C. The results are shown in fig. 9 together with results of measurements on pure Al[8] and extruded SAP 960[9]. From the figure it appears that whereas the activation energy as determined by tensile testing of pure Al above 300°C is constant to about 35 kcal/mole, the energies of the three Al/Al_2O_3 alloys increase with increasing temperature over 300°C, independently of grain size.

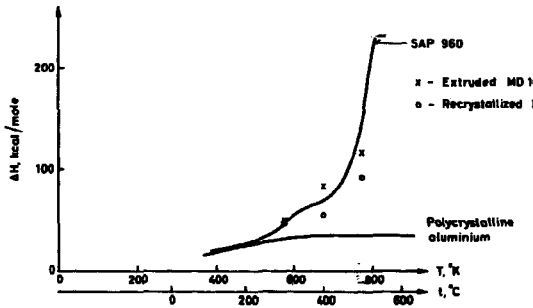

Fig. 9. Activation energies in tensile testing of aluminium alloys.

Irradiation Experiments

Irradiation Effects in SAP[10]

Examination of tensile, fatigue and impact specimens of SAP 930, SAP 895 and SAP 865 irradiated at 400°C with a dose of approximately 5×10^{20} n/cm^2 (E > 1 MeV) was continued.

Tensile specimens machined from flash-welded SAP 930 and SAP 895 rods were irradiated in the DR3 reactor at 400°C with a dose of about 5×10^{20} n/cm^2 (E > 1 MeV). Testing and examination of the flash-welded specimens

7) Sherby et al., Acta Met. 5, 219 (1957).

8) Trozera et al., Trans. ASM 49, 173 (1957).

9) P. Guyot, Eur., 550 ff. (1964).

10) Metallurgy Department Annual Progress Report for the Period Ending 31st March, 1965. Risö Report No. 110, 21-23 (1965).

will conclude the Risö SAP irradiation and examination programme, and a final report on the effects of fast-neutron irradiation on the mechanical properties of commercial SAP alloys is planned to be issued soon.

Pressure-Vessel Steel Irradiations

This programme comprises three high-strength development steels ($\sigma_y \geq 60$ kg/mm^2) and an A302B type steel as a reference ($\sigma_y = 45$ kg/mm^2). Nominal compositions and room-temperature mechanical properties of the four steels are given in table III.

The 248 SV steel has an austenitic and martensitic structure in the hardened and tempered condition. The structure of the 2 RMO steel is similar, though without any ferrite. The BH 70 steel is fully hardened; in thick plates the outer layers are of martensitic structure whilst the interior is bainitic.

Irradiation of charpy, tensile and miniature impact specimens at three temperatures between 200 and 300°C with doses of 10^{18} to 10^{20} fast neutrons per cm^2 will be carried out in the DR3 reactor at Risö and the R2 reactor at Studsvik. A total of 23 irradiations have been planned.

Table III

Nominal Composition and Room-Temperature Mechanical Properties of High-Strength Potential Pressure-Vessel Steels

	Nominal Chemical Composition													Room-temp. mechanical properties			
Material designation / Manufacturer	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	V	N	Al	Ti	Yield strength kg/mm^2	UTS kg/mm^2	Elongation %	Impact strength -20°C kg/mm^2
248 SV Avesta Sweden	.04	.53	.76	.02	.01	16.0	4.70	1.20	-	-	.04	-	-	80	85	18	18
2 RMO Bofors Sweden	.07	.44	.66	.07	.07	12.9	5.3	1.5	-	-	-	-		74	92	20	12
BH 70 Henrichshütte W. Germany	.16	.30	.30	.02	.02	.30	3.3	.38	.02	.04	-	-	.02	67	80	16	12
DE 631A Degerfors Sweden	.19	.29	1.46	.01	.02	.06	.07	.45	.16	-	.01	.03	-	47	64	-	10

Irradiation Growth of Dilute Uranium Alloys

The experimental part of this programme was finished by March 31st of this year. The major results of the metrology, density, metallography, and hardness measurements of the irradiated cylindrical specimens (5 mm Ø by 50 mm long) will be presented at the Institute of Metals Spring Meeting in London this year. The overall conclusions that can be drawn after a preliminary evaluation of the data are:

(a) Adjusted (Fe, Al) uranium specimens containing a small amount of [010] and [100] texture grow in the direction of the [010] texture and shrink in the direction of the [100] texture when irradiated at 260°C.

(b) The growth/shrinkage increases at a linear rate with burn-up in the zero to 1000 MWD/te range.

(c) In the 1000 to 2000 MWD/te burn-up range a growth saturation takes place, and from 2000 to 4000 MWD/te burn-up no further irradiation growth is seen.

(d) In the growth saturation range, 1000 to 2000 MWD/te, also the metallographic structure is altered. It becomes mottled and smeared out, and the original grains can hardly be distinguished by polarized-light examination.

(e) At low burn-up, zero to 1000 MWD/te, the hardness increases steeply with burn-up, from 280 to 400 kg/mm^2. Between 1000 and 4000 MWD/te the hardness increase is much slower.

(f) Surface roughening is severe even at 500 MWD/te burn-up. The coarseness of the roughening pattern agrees quite well with the pre-irradiation grain sizes of the specimens.

(g) Specimens of adjusted (Fe, Al) uranium alloyed with 5/8 a/o molybdenum, isothermally transformed, show negligible dimensional changes at 2000 to 3000 MWD/te burn-up, but surface roughening is very considerable, conforming with the very large grain size in this material. Since the individual grains develop irradiation growth whilst the overall dimensions of the specimens do not change, the material must be essentially texture free.

(h) Also, specimens of cast uranium 3 a/o molybdenum alloy are dimensionally stable after 2000 to 3000 MWD/te burn-up. The surface roughening of this material is very slight, conforming with its very small grain size.

(i) The irradiated specimens show surprisingly small density decreases, the magnitude being hardly more than expected from solid fission products alone.

Irradiation of Uranium-Dioxide/SAP Fuel Pins[*]

Post examination has been started. So far, all pins have been removed from the capsules in which they were irradiated in a lead bath. Eight of the ten pins were without any detectable fissures in the SAP cladding whereas the last two pins were damaged extensively. The reason for the damage is believed to be that during start-up of the reactor, when the lead was still solid, these pins could not expand freely in the capsules as their tops touched the capsule lid.

Rig II of this series was irradiated to a burn-up of 3940 MWD/t UO_2 (average over all capsules). Because of a leak in the cooling system of the rig, irradiation had to be stopped and the rig to be removed from the reactor before the scheduled average burn-up of 5000 MWD/t UO_2, was reached. Post examination of this rig has also been started. So far, the capsules containing the SAP-UO_2 pins have been removed from the rig and have been visually inspected and X-rayed. Defects of the type observed in the two defective pins from rig I have not been detected by these examinations.

Development of Post-Irradiation Examination Techniques

The projects initiated and discussed in last year's progress report progressed, and additional techniques are being developed.

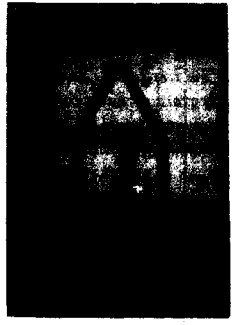

Fig. 10. Universal measuring jig for determining dimensional changes of irradiated fuel specimens.

[*] This programme has been carried out together with the Ceramics Group.

Metrology

The measuring jig, fig. 10, accommodating specimens of up to 400 mm length and 40 mm diameter, was constructed. It has provision for measuring overall length, diameter and bow at any length position and any rotational position of the specimens. After out-of-cell commissioning work, some modifications have been incorporated.

Leak testing of irradiation capsules, fuel rods, etc.

The bubble leak-detection apparatus was commissioned in the summer of 1965 and has been used to test about 40 irradiation capsules and fuel pins. It has been found to give a clear indication of size and position of holes down to a minimum size of 5 microns, and a good permanent record of the leaks is obtained by photographing the emergent bubble streams through the cell window and the window in the bubble chamber, as illustrated in fig. 11.

Since the present prototype apparatus has a length limit of 300 mm for specimens to be tested, design has started on a 1 metre long leak testing chamber, which will allow fuel rods from the Halden irradiations to be tested.

Fig. 11. Irradiated fuel specimen showing gas-bubble streams from two leaks.

X-radiography of fuel rods, fuel specimens, etc.

The X-radiography facility was commissioned in the autumn of 1965, and radiographs of reasonably good quality have been obtained. Special commercial X-ray films with reduced gamma-irradiation sensitivity are used so that serious gamma fogging is avoided. For the radioactivity levels so far encountered in items to be radiographed it has not been necessary to resort to special film processing techniques, but it may be necessary at a later date.

Easy and accurate positioning of specimens in relation to the X-ray beam and the film is obtained by the use of a specimen holder, which can

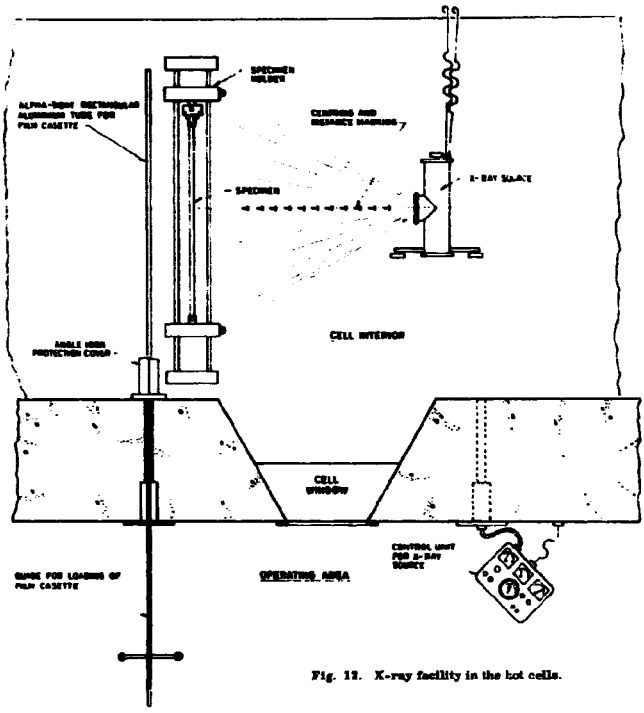

Fig. 12. X-ray facility in the hot cells.

accommodate specimens ranging in length from a few to 150 cm. Since the specimens are held in a lathe-type chuck, radiographs can be obtained for any desired rotational position. The width of specimens is limited by the film cassette to 70 mm. The arrangement of the whole X-radiography facility is illustrated in fig. 12.

Fission-gas sampling

The equipment for puncturing of irradiation capsules and fuel rods, pressure and volume measurements and sampling for mass-spectrometric analysis was manufactured and assembled.

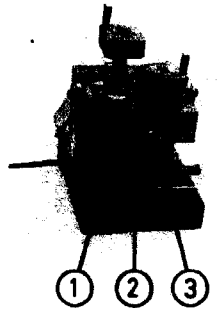

Fig. 13. Piercing unit from apparatus for fission-gas sampling.
1: piercing valve; 2: specimen; 3: specimen holder.

The design of the piercing unit is illustrated in fig. 13. The main part of the unit, a modified bellow-type vacuum valve, is screwed down so that it is sealed to the specimen via an O-ring. By further tightening of the screw the resistance of a coil spring is overcome so that a needle moves relative to the mouth of the unit, and the piercing takes place.

Impregnation of metallographic specimens

The technique of immersing a section of a ceramic fuel rod in cold-setting resin and evacuating the air from the specimen via the resin, followed by the return of the system to atmospheric pressure, was found to give only incomplete filling of the crack networks. A substantial improvement was obtained by evacuating the specimen and the resin separately before immersing the former into the latter.

METAL AND CERAMICS TECHNOLOGY

Metal Technology

The major part of the work was devoted to fuel-element development. Two types were studied, namely an aluminium plate element (MTR-element) for the DR3 reactor at Risö and a zircaloy/uranium-dioxide power-reactor fuel element to be installed in the Halden reactor in the summer of 1967.

This work was carried out in close collaboration with staff from the Elsinore Shipbuilding and Engineering Co., Ltd., seconded to the Department.

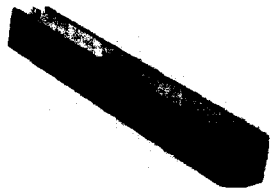

Fig. 14. Fuel box for MTR fuel element.

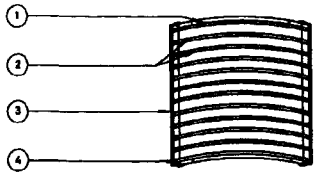

Fig. 15. Cross section of fuel box. 1: upper cover plate; 2: fuel plates; 3: side plate; 4: lower cover plate.

MTR Fuel Elements

Figs. 14, 15 and 16 show the design of the plate-type fuel element.

The fissile material is located in the fuel box (fig. 14), which consists of ten fuel plates and two cover plates (fig. 15). The fuel plates and the aluminium cover plates are located in milled tracks in the side plates.

Welded onto the fuel box are an inlet and an outlet cave as well as a tube assembly to ensure correct placing in the reactor core. Uranium metal (93% enrichment) is placed in the fuel plate, which consists of a core of uranium-aluminium alloy with 20 wt.% uranium, surrounded by an aluminium frame and provided with aluminium cover plates on both sides (fig. 16).

The fabrication of the plate-type fuel element takes place in three steps:

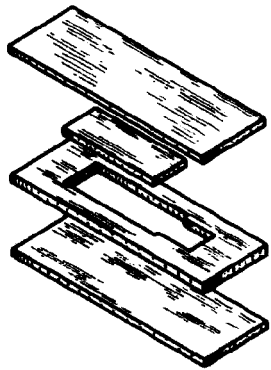

Fig. 16. Components of MTR composite fuel plate.

(1) casting of uranium-aluminium alloy for core material in the fuel plate;
(2) rolling of the fuel plate;
(3) assembly of the fuel element.

(1) The uranium-aluminium alloy with 20% uranium is melted and cast in a high-frequency vacuum furnace. The casting procedure was developed with aluminium/natural-uranium alloys.

The question is to get a homogeneous distribution of uranium in the slab. The distribution is controlled by X-ray examination, and the experiments indicated that the casting temperature and the cooling rate of the slab must be very carefully controlled for a satisfactory uranium distribution to be obtained.

(2) The slab is hot rolled to a suitable thickness for punching the core piece. The aluminium frame round the core piece is punched with the same tool as the core piece.

The frame and core pieces are covered with aluminium plate on both sides and hot and cold rolled to the final thickness and dimensions of the fuel plate.

(3) Assembly of the fuel element is carried out by pinning and the mechanical stability ensured by welding reinforced Al strips on the element.

The first element was placed in the core of DR3 in December 1965, and two more were added in January and February 1966. All elements behaved satisfactorily, and after irradiation in DR3 the elements will be examined in the hot cells.

Fig. 17. Instrumented fuel plate for test section for D_2O-loop boiling experiment.

D_2O-Loop Boiling Experiment

Concurrently with the fabrication of MTR plate elements, test sections for a heavy-water loop in DR3 were made. In this experiment the boiling of the heavy water on the surface of an instrumental fuel plate will be studied, and a test series is planned to end with melting of the fuel plate (see fig. 17). The internal dimensions of the cooling channel are very critical, and an eddy-current technique for the control of this channel has been developed in collaboration with the Danish Central Welding Institution in Copenhagen.

The small dimensions of the fuel section and the heavy instrumentation have posed several problems in the manufacturing process, especially in welding. These problems have been solved, partly in collaboration with the Danish Central Welding Institution, and the first section has been delivered to the reactor.

Uranium-Dioxide/Zircaloy Fuel Elements

In order to examine the problems connected with the fabrication of power-reactor fuel elements consisting of UO_2 in zircaloy-2 tubes, test welding by the TIG method was carried out on zircaloy-2 tubes and end caps.

The welding was made in helium and controlled automatically. The end closure weld was subjected to X-ray examination, helium leak test, corrosion test, and metallographic examination.

Fig. 18. Arrangement for automatic welding of zircaloy-2 fuel rods.

Fig. 18 shows the equipment used in the He-filled chamber during the first welding trials.

The materials (zircaloy-2 tube and rod) used in the fabrication of fuel pins were examined for defects at the Danish Central Welding Institution by ultrasonic inspection. To increase the inspection speed, an eddy-current technique is being developed. The wall thickness of the tubing is measured by an ultrasonic technique, and the inner diameter is controlled by an air-gauge method.

The different non-destructive methods have been developed as a part of the SAP programme[11], and it has been possible by minor changes to adapt the methods for the zircaloy-2 material.

11) Risø Report No. 110 (1965).

Fissionable Material Inventory System

In the manufacture of MTR plate elements and power-reactor fuel pins, large amounts of fissionable material will be in production. Therefore an inventory system has been established for the Metallurgy Department to control the amount (with an accuracy of 0.1 g) and location of fissionable material. New regulations will be built into the system to avoid criticality.

Welding of Metal-Sheathed Thermocouples

The development of methods for welding steel-encapsulated thermocouples was continued, and about 1800 couples were manufactured for the departments of Risö and for various industrial concerns.

Powder Metallurgy

Creep Properties of Aluminium/Aluminium-Oxide Products Made from Atomized Aluminium Powders

Extruded rods were manufactured from atomized aluminium powders having average grain sizes of 22, 8.4 and 4.0 microns. The oxide contents of the powders are given in table IV together with their surface areas. The oxide phase is present as natural oxide on the particles, and no oxide has been blended in. Rod specimens with their longitudinal axis in the extrusion direction were creep tested at 400°C.

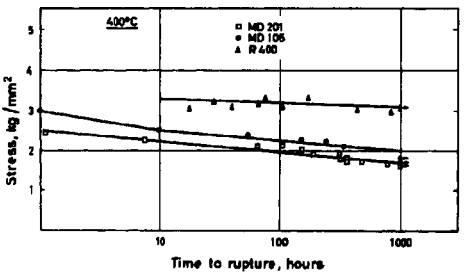

Fig. 19. Rupture lives of experimental aluminium-oxide alloys made from atomized aluminium powders.

The rupture lives of the materials are given in fig. 19, which shows good high-temperature strength even for MD 201 containing only a small amount of oxide. The strength increases with decreasing particle size of the base material because of the higher concentration of dispersed oxide particles acting as barriers to dislocation movement.

The total and uniform creep elongation is plotted in figs. 20 and 21. For the fine powders MD 105 and R 400 the uniform elongation after about 1000 hours' creep decreases to about one per cent. Products made from MD 201 were expected to have a higher ductility on account of the smaller content of aluminium oxide; the elongation data for this material were scattered between 0.3 and 2 per cent.

On the basis of these experiments it is concluded that even the presence of a small amount of an aluminium-oxide phase, finely distributed in aluminium, decreases the ductility at elevated temperatures quite drastically in comparison with pure aluminium.

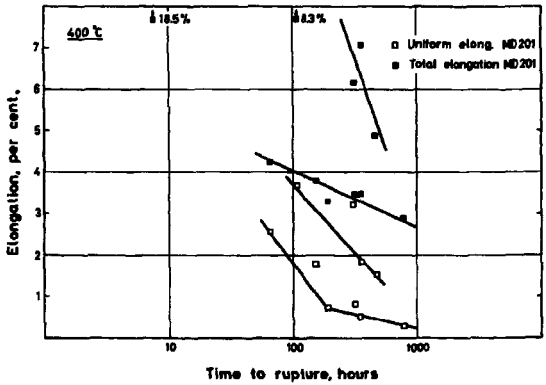

Fig. 20. Elongation of experimental aluminium/aluminium-oxide alloys made from MD 201 atomized aluminium powders.

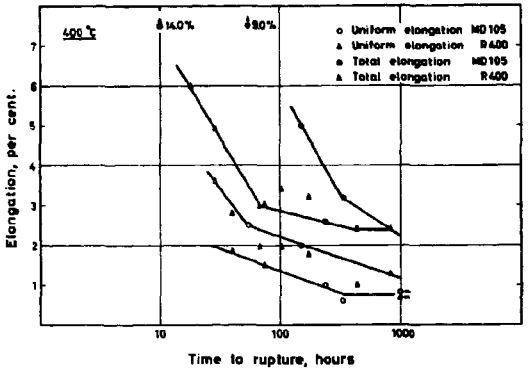

Fig. 21. Elongation of experimental aluminium/aluminium-oxide alloys made from atomized aluminium powders.

Table IV

Raw Materials

Powder	Supplier	Sedimentation surface area m^2/g	Average particle diameter microns[a]	Oxide content weight %
MD 201 aluminium[b] (< 40 microns)	Metals Disintegrating, U.S.A.	0.10	22	0.3
MD 105 aluminium	"	0.35	6.4	0.6
R 400 aluminium	Reynolds Metals Co., U.S.A.	0.56	4.0	1.0
P 110 C1 aluminium oxide	Degussa, Germany	24.5	0.078	-

[a] Diameter of uniform spheres corresponding to the sedimentation surface area.

[b] The commercial powder has been sieved before application.

Creep Properties of Blended Aluminium/Aluminium-Oxide Powder Products

The creep properties of aluminium/aluminium-oxide products made by the powder blending technique were examined at 400 and 500°C. The products were made from R 400 and MD 201 atomized aluminium powder with an admixture of P 110 Cl aluminium-oxide powder. For characteristics of these powders see table IV.

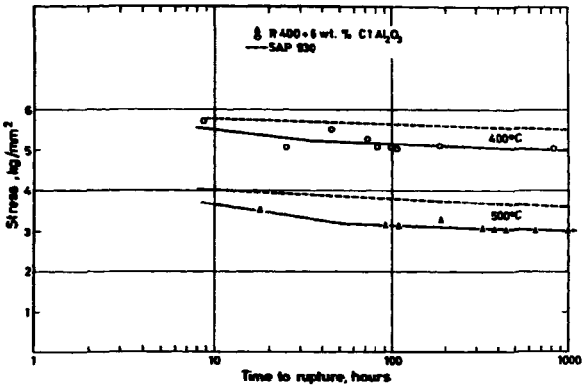

Fig. 22. Rupture lives of experimental aluminium/aluminium-oxide alloys containing 6 weight per cent aluminium-oxide and SAP 930.

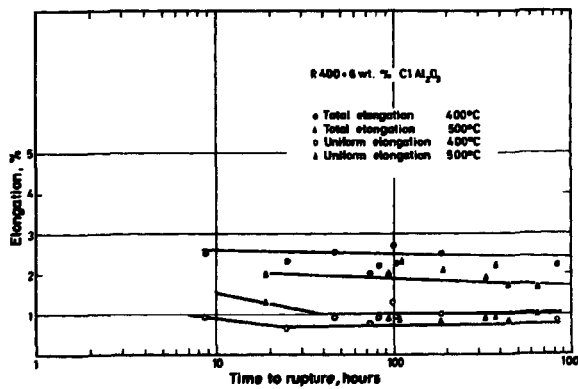

Fig. 23. Elongation of experimental aluminium/aluminium-oxide alloys containing 6 weight per cent aluminium oxide.

The rupture lives of the products made from R 400 aluminium powder containing six weight per cent aluminium-oxide are shown in fig. 22 together with the rupture life of a commercial SAP 930 containing approximately seven weight per cent aluminium oxide. The strength properties of the two types of products are comparable. The total and the uniform elongation of the powder-blended products are shown in fig. 23. The elongation values are higher than those found for SAP 930 (see Geneva paper A/Conf./28/P/421), but as the powder-blended products have only been manufactured on a laboratory scale, such results may only be considered indicatory.

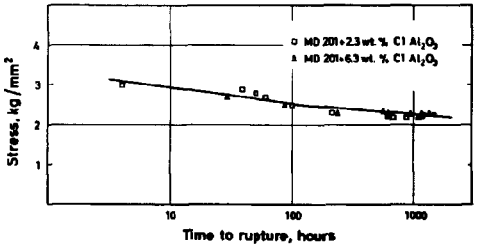

Fig. 24. Rupture lives of products made from coarse-atomized MD 201 powder containing 2.3 and 6.3 weight per cent oxide added by blending.

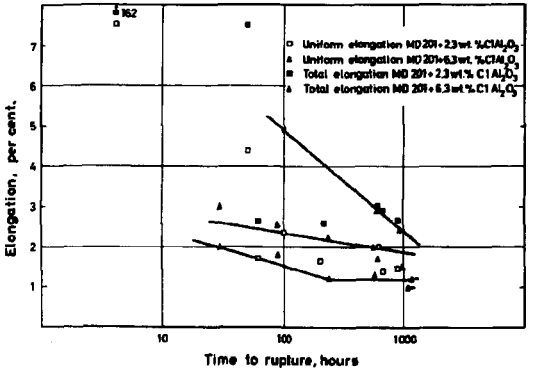

Fig. 25. Elongation of products made from coarse-atomized MD 201 powder containing 2.3 and 6.3 weight per cent oxide added by blending.

The rupture lives of the products made from the coarse-atomized powder MD 201 with 2.3 and 6.3 weight per cent oxide blended in are shown in fig. 24. The increase in oxide addition from 2.3 to 6.3 per cent by weight has no effect on the strength, which is in agreement with microscopic analysis, indicating that most of the oxide is present as coarse agglomerates.

The total and the uniform elongation of the powder-blended products are shown in fig. 25. An increase in the amount of oxide admixture from 2.3 to 6.3 per cent decreases the ductility. Thus, if we consider strength and ductility data together, no gain has been obtained by increasing the amount of oxide to 6.3 per cent.

A comparison of the creep properties of the powder-blended and atomized-powder products described above indicates that addition of oxide to the atomized powders may increase the strength of the final products significantly without having much influence on their ductility. For the R 400 products an oxide addition of six per cent by weight increases the strength, causing rupture in 1000 hours at 400°C from 3.0 to 5.0 kg/mm^2, and reduces the corresponding uniform elongation from about one to about 0.8 per cent. For the powder MD 201 an oxide addition of 2.5 per cent increases the strength, causing rupture in 1000 hours at 400°C from 1.7 to 2.5 kg/mm^2. A comparison of elongation values cannot be made on the basis of the present data because of the great scatter in the results for the MD 201 atomized-powder products. The uniform elongation of the powder-blended MD 201 products for a time to rupture of 1000 hours is in the range 1.0 to 1.5 per cent.

If we compare the powder-blended products made from R 400 and MD 201 atomized powders containing respectively 6.0 and 2.3 per cent oxide, it is seen that the small gain in uniform elongation shown by coarse MD 201 products is obtained at the expense of a great reduction in strength. It is therefore concluded on the basis of these results, which are to be considered indicatory only, that the R 400 products with about 6 per cent oxide blended in have the best combination of strength and ductility at 400°C.

Micronizing and Air Separation of Aluminium Powder*

A system for micronizing and separation of powder has been built, see fig. 26.

The apparatus consists of a fluid energy jet-O-mizer mill 0101, four cyclones in series and a hose filter.

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* This work has been carried out in collaboration with J. G. Rasmussen, D. Sc., Department of Metallurgy, The Technical University of Denmark.

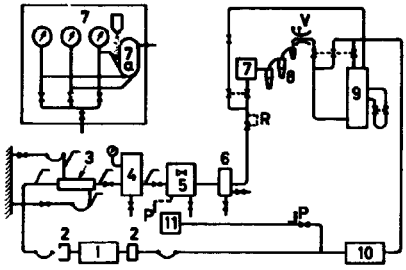


Fig. 26. Micronizing separation system. 1: Compressor; 2: Pressure variation vessel; 3: Heat interchanger; 4: Reservoir; 5: Oil-water filter; 6: Dust filter; 7: Jet-O-Mizer stand; 7a: Jet-O-Mizer; 8: Cyclones; 9: Cyclone-hose filter; 10: Filter; 11: Filter; P: pitometer; R: reduction valve; V: Venturitube

The air flow is 100 m^3/h, the pressure at the mill 7.2 atm. The air accelerates through nozzles, and the speed of the stream in the mill is supersonic. Between the compresser and the mill the air is cooled and filtered; it is then dried and cleaned of oil vapour and dust from the previous filters, so that contamination of the powder cannot take place. To extend the lifetime of filters and to make possible the use of inert gases, recirculation is used. It is possible to control the efficiency of the cyclones so that desired fractions of powder can be manufactured by changing the inlet speed. This is done by means of two Pitot tubes and a Venturi tube.

The hose filter is cleaned by reversing the air flow through the filter housing. The deposits on the filter surface are blown down into the reservoir under the fourth cyclone, which is built into the filter housing. The reversing of the air stream is controlled by seven ball valves which are air-pressure operated from one single valve.

The apparatus is finished (see fig. 27), and the introductory micronizing experiment has begun.

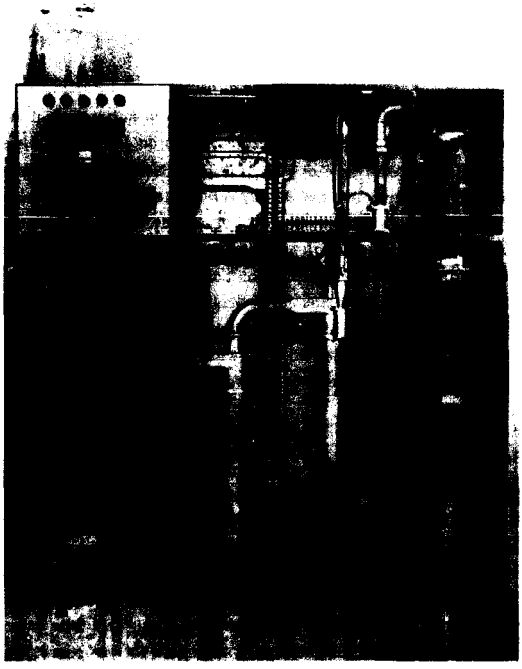


Fig. 27. Apparatus for micronizing separation.

Determination of Grain Size of Aluminium Powder

Sedimentation analysis in chloroform

Sedimentation analysis of very fine powder (0.3-3 microns) in soapy water[12] is impossible because of the reaction of the powder to the soapy water. This difficulty can be overcome by using cyclohexanol as sedimentation medium, a method which, however, is very slow because of the high viscosity of cyclohexanol. A third method has therefore been developed, using chloroform as the sedimentation medium. The analysis time is reduced because of the lower viscosity, and the powder is not so liable to

12) Risö Report No. 110 (1965).

agglomerate in chloroform as in soapy water. Results from analyses of a coarse powder (1-10 microns) show good agreement with those previously obtained with soapy water and cyclohexanol.

Ceramics

Preparation of Uranium-Dioxide Pellets

A new method was developed for precipitation of ammonium diuranate (ADU), as it soon became evident during the scaling up of the pellet-production unit that the filtering properties of ADU precipitated by the method previously adopted[13] were insufficient for large-scale production. After a study of the influence of the precipitation conditions on the filtering properties of ADU, the two-step continuous precipitation method developed in France[14] was finally adopted. After a thorough investigation, the optimum precipitation conditions yielding an ADU with good filtering properties from which UO_2 pellets with high density can be prepared were found to be

First step,	pH = 3
Second step,	pH = 7
Capacity	20 kg/24 h.

Calcination and reduction experiments were performed to find a suitable method for fabrication of a stable UO_2 powder which oxidizes only slightly during storage in air. The investigation showed that a stable powder can be obtained when the powder is calcined in air or in nitrogen at 700°C before reduction is performed, likewise at 700°C. This treatment has therefore been adopted for the small-scale powder-preparation unit developed, and a continuous calcination furnace is under construction.

During the fabrication of pellets from ADU precipitated by the two-step method mentioned it became evident that the pellets sintered with a rather rough and irregular surface and that after grinding they contained a substantial number of surface porosities. Vacuum drying and sieving of the binder (zinc stearate) decreased this number considerably, but still the pellet surface after sintering was not satisfactory. An examination of the UO_2 powder showed that it contained a few per cent coarse granules which could not be crushed by the pressing. Different methods of removing this fraction (sieving, ball milling and hammer milling) were examined, and it appeared that high-speed hammer milling was the most efficient and economical

13) Risö Report No. 90 (1964).

14) Bull. d'information scientifique et technique 80 (1964) 20.

method of overcoming this problem. A continuous high-speed hammer mill was constructed, and the method has been adopted for the pellet-production unit.

Investigation of Pellet-Pressing Techniques

An investigation of different techniques for pressing of UO_2 pellets has been started. The objective is to define the difference between various techniques (forced and frictional die movement) and to find a method by which pellets can be pressed and sintered to close tolerances so that grinding of the pellets before use can be omitted. The hydraulic press normally employed for UO_2 pellets has been equipped with pressure transducers to record the pressure and the counter pressure from the die, and with a mechanical transmission with which the velocity of the upper plunger and the die can be measured and recorded. Several experiments have already been performed, and although the investigation is not yet completed, it has been possible from the results obtained to define pressing conditions giving a satisfactory double pressing.

Irradiation of Uranium-Dioxide Pellets Prepared at Risö[*]

Rigs II and III were irradiated, and the post examination was started with removal of capsules from the rigs, leak testing and X-raying of the capsules. The rigs had to be removed from the reactor before the scheduled date because of a sudden increase of activity in the rig atmosphere. An examination of the capsule indicated that the leakage was due to overheating of the capsule, which could be explained by a slight displacement of the pellets in the capsule in combination with a critical position of an insulation gap in the rig.

Rig II was equipped with the fission-gas detector described previously. To measure the fission-gas pressure as directly as possible the detector was placed between the two capsules. Unfortunately, the γ-heating of the detector in this position became excessive as the detector was placed in the reactor core itself, and it stopped functioning when 7 MW was reached during the first start-up of the rig. Rig IV of this series, which was started up on the 1st February, 1966, was also equipped with a fission-gas detector, but in this rig the detector was moved to the upper part of the rig thimble, where the γ-flux was expected to be low. Until now, the detector has functioned satisfactorily.

[*] This programme has been carried out together with the Irradiation Experiment Group.

Both in rigs II and IV, tungsten/tungsten-rhenium thermocouples were placed in the centre and chromel/alumel thermocouples near the surface of the pellets. Valuable information about the temperature distribution in the pellets was obtained from these thermocouples although the centre ones lasted only for short periods after the start-up.

EDUCATION AND TRAINING

The educational activities arranged for the benefit of the staff included seminars and courses of metallurgy organized by Professor J. Anderson, San Jose State College, at present attached to Risö. Also study-group work in the fields of dislocation theory and computer techniques was carried out.

Members of the scientific staff took part in the teaching at institutes of higher education. N. Hansen and K. Rørbo taught the science of engineering at the Danish Academy of Engineering, and P. Knudsen lectured on vacuum technology at the same academy. H. E. Gundtoft lectured on the metallurgy of steel in a refresher course arranged by the Institute of Technology in Copenhagen. One student from the Technical University of Denmark and three from the Danish Academy of Engineering worked in the department in preparation for their final examination. Two postgraduate students, T. Nilsson and H. Lilholt from the Technical University are working in the department on the completion of their theses. Finally, J. Balling Jensen, A. Grum-Jensen, H. E. Witthøft, and H. Hougård, are attached to the department on grants from the Danish A. E. C.

PUBLICATIONS AND INTERNAL REPORTS

External Publications

S. A. Lund (Danish Welding Institution): P. Knudsen:	Non-destructive Inspection Methods Applied to Multifinned SAP Tubing for Nuclear Fuel Elements. Paper read at the IAEA congress on Non Destructive Testing in Nuclear Technology, Bucharest, May 17-21, 1965.
N. Kothari:	Sintering Kinetics of Tungsten in the Presence of a Liquid Phase. Trans. Ind. Inst. of Met. 17 (1964) 199-210.
T. Lauritzen: P. Knudsen:	The Reaction of Sintered Aluminium Products with Uranium Dioxide and Monocarbide. J. Nucl. Mat. 16 (1965) 173-183.
A. C. Winther:	Facilities for Mechanical Testing of Irradiated Reactor Materials at Risö. Risö Rep. No. 98 (1965).
N. Kothari:	The Effect of Particle Size on the Sintering Kinetics in Alumina Powder. J. Nucl. Mat. 17 (1965) 43-53.
N. Hansen, H. Lilholt and M. Jensen:	Bibliography on Dispersion-Strengthened Materials. Risö Rep. No. 48, Supplement 2.
O. Toft Sørensen:	Fremstilling og undersøgelse af uran-dioxid reaktorbrændsel på Risø (Preparation and investigation of uranium-dioxide reactor fuel at Risö). Ingeniøren 20 (1965) 647-654. (In Danish only.)
O. Toft Sørensen:	Kontinuert højtemperatur sintringsovn på Risø (Continuous high-temperature sintering furnace at Risö). Ing. Ugeblad 10 (1966) 24-25. (In Danish only.)

B. Vigeholm: The Effect of Solute Concentration on the Formation of Loops in Al-Mg Alloys during Fission Fragtment Irradiation. Phys. Stat. Sol. 12 (1965) 877.

Internal Reports 1/4-65 - 1/4-66

(Not generally available. Requests for information concerning these reports may be addressed to Mr. Niels Hansen, M. Sc., Head of the Metallurgy Department Research Establishment Risö, Roskilde, Denmark.)

No. A-77
J. Christensen: Brazing of Cooling Jackets for Water Cooled 7V Irradiation Rigs. 19 pp.

No. A-78
J. Christensen: Udvikling af Ovn af Rustfast Stål med påloddet varmelegeme for fissil Rig (Soldering of heating element to stainless-steel furnace for fissile rig). 5 pp. (In Danish only.)

No. A-80
T. Lauritzen:
P. Aastrup: The SAP-UO_2 Irradiation Experiment Design and Fabrication of Fuel Pins for Rig I. 22 pp.

No. A-82
E. Adolph: The Effect of Heat Treatments on the Structural Stability of the Sintered Aluminium Products Designated SAP-930-S-4M and SAP 930-V. 8 pp.

No. A-86
H. Lilholt: Investigation of the Relation between Magnification and Specimen Position in the Electron Microscope. 10 pp.

No. A-85
E. Adolph: Failure in a SAP 895 Tube Used in Static Tests at 300° to 400°C with Internal Pressures from 10 to 15 kg/cm^2 Provided by OM 2. 10 pp.

No. A-89
K. Rørbo: Stability of Stainless Steel Sheathed Thermocouples in Molten Lead. 4 pp.

No. A-91
N. Kothari: Some Physical Properties of Ball-Milled Aluminium Powder Alloys. 14 pp.

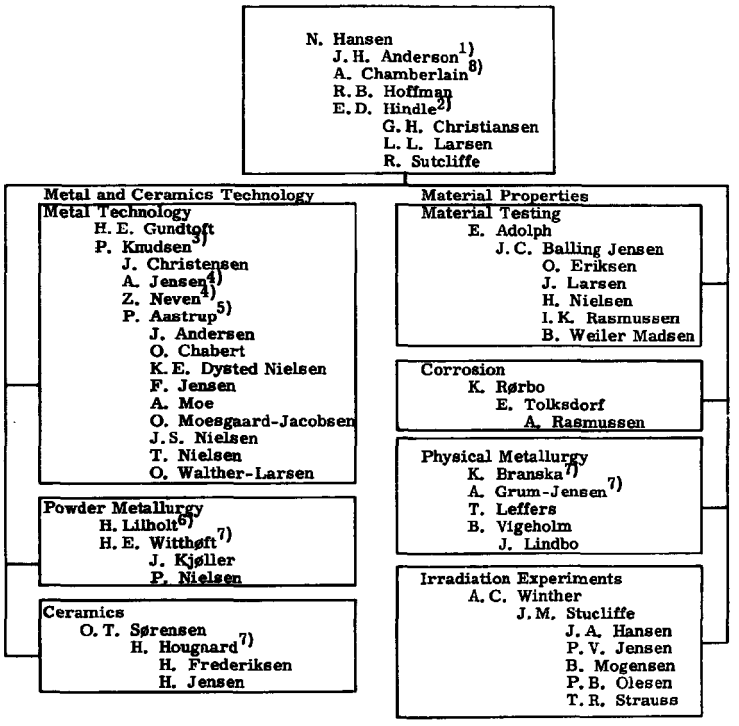
No. A-90 P. Aastrup: A. Moe:	Welding of Stainless Steel Capsules for UO_2 Irradiation Experiments. Establishment of Procedure. 16 pp.
Nos. M-1017 and M-1018 K. Rørbo: B. Skytte Jensen, (Chemistry Dept.): S. O. Nielsen (Chemistry Dept.):	Anvendelse af opløst borsyre til kontrol af DK 400 (Application of boric-acid solution for control of DK 400). (In Danish only.)
No. A-96 R. B. Hoffman:	Fissionable Material Inventory System for Metallurgy Department. 9 pp.
No. A-95 H. Lilholt:	Separation of Aluminium Powder by Centrifugal Sedimentation. 29 pp.
No. A-94 K. Rørbo:	Investigation of a Corroded DR2 Fuel Element. 11 pp.
No. A-99 N. Kothari:	Mechanical Properties of Ball-Milled Aluminium Powder Alloys. 6 pp.
No. A-106 N. Kothari:	Physical and Mechanical Properties of Sub-Oxidized Ball-Milled Aluminium Powder Alloys. 10 pp.
No. A-107 N. Kothari:	Recovery and Recrystallization of Quenched Al/Al_2O_3 Alloys. 7 pp.
No. A-105 O. Toft Sørensen:	Bestrålingsforsøg med UO_2-piller. Status pr. 15/2-66. (Irradiation experiments with UO_2-pellets). 16 pp. (In Danish only.)

PAPERS AND ORAL PRESENTATIONS GIVEN AT TECHNICAL MEETINGS

(not available)

Z. Neven:	Brændselselementer (Fuel elements). Meeting about the reactor development work of the Danish AEC, October 1965.
N. Hansen:	Extrusion af sintrede aluminiumlegeringer (Extrusion of sintered aluminium alloys). The Danish Metallurgical Society, December 1965.
T. Leffers:	Textur belyst teoretisk og praktisk (Texture, theoretically and practically). The Danish Metallurgical Society, December 1965.
T. Nilsson:	Duktilitet af en aluminium/aluminium-legering (Ductility of an aluminium/aluminium-oxide alloy). The Danish Metallurgical Society, December 1965.
Z. Neven:	Valsning af compound-plader til nukleare brændselselementer (Rolling of compound plates for nuclear fuel elements). The Danish Metallurgical Society, December 1965.
Prof. J. H. Anderson, San Jose State College, U. S. A.	The Relaxation Spectrum. The Danish Metallurgical Society, December 1965.
B. Vigeholm:	Punktdefekter i metaller (Point defects in metals). Dansk Metallurgisk Selskab, 15/2-66. Report: Ingeniørens Ugeblad, 8/4-66.
K. Rørbo:	Korrosion og vandkemi i kraftreaktorer (Corrosion and water chemistry in power reactors). Risø, February 1966.

ORGANIZATION CHART



1) Professor on sabbatical leave from the San Jose State College, U. S. A.

2) On leave from the U. K. A. E. A., Springfields Laboratories, England.

3) At present at MIT, U. S. A.

4) On leave from the Elsinore Shipbuilding and Engineering Co., Ltd.

5) On leave from the Danish Central Welding Institution.

6) Postgraduate student at the Technical University of Denmark.

7) A. E. C. research fellow.

8) On leave of absence from the U. K. A. E. A.